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**LARC-TPI: A MULTI-PURPOSE THERMOPLASTIC
POLYIMIDE**

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Anne K. St. Clair and Terry L. St. Clair

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National Aeronautics and
Space Administration

Langley Research Center
Hampton, Virginia 23665

INTRODUCTION

Linear aromatic polyimides are presently under consideration for applications on future aircraft and spacecraft. As a class, linear polyimides are attractive to the aerospace industry because of their toughness and flexibility, remarkable thermal and thermo-oxidative stability, radiation and solvent resistance, low density, and excellent mechanical and electrical characteristics over a wide temperature range. However, the processing of these materials is tedious compared to other engineering plastics. A soluble polyamic acid precursor must first be dissolved in a high-boiling solvent, and later converted to the polyimide by heating to a high temperature which causes evolution of water and residual solvent. The preparation of void-free moldings, large-area laminates or adhesive joints with such a material becomes exceedingly difficult.

The use of meta-substituted aromatic diamines in the preparation of linear polyimides has been a major advancement toward improving the processability of these polymers and lowering the glass transition temperature (1). A further advancement has been the synthesis of these polyimides in a nontoxic, low-boiling ether solvent (2). The adhesive lap-shear strength of a meta-oriented polyimide was improved 2 to 5-fold by synthesis in bis(2-methoxyethyl)ether (diglyme) (3,4). More recently, this material was used as an adhesive for bonding ultrathin polyimide film in the proposed NASA-Solar Sail Program (5). The small-area sail joints (0.64 cm overlap) allowed an easy escape for volatiles during processing. However, the need still exists for a high-temperature adhesive that can bond larger areas without the evolution of volatiles and the entrapment of voids.

LARC-TPI is a linear thermoplastic polyimide which is presently being developed at NASA-Langley Research Center as an adhesive for the large-area bonding of metals and films. This material is a linear polymer which contains

a meta-substituted diamine and is prepared in an ether solvent. Unlike conventional polyimides, LARC-TPI can be imidized and freed of volatiles at a reasonably low temperature (230°C) and then be processed as a thermoplastic. In an effort to exemplify the multiple uses of this material, this paper reports the preliminary development of LARC-TPI as an adhesive, molding powder, composite matrix resin, high-temperature film and fiber.

EXPERIMENTAL

Preparation of LARC-TPI

The monomers used in the preparation of LARC-TPI were 3,3',4,4'-benzophenone tetracarboxylic dianhydride (BTDA) and 3,3'-diaminobenzophenone (3,3'-(DABP). The BTDA was used as received from Gulf Chemicals*, m.p. 215°C, after drying overnight in a vacuum oven at 140°C. The 3,3'-DABP was obtained from Ash Stevens, m.p. 150°C, and used as received.

The polymerization was performed in a closed vessel at 20°-25°C at a concentration of 15% solids by weight in reagent grade diglyme. The 3,3'-DABP (21.2 grams) was slurried in 302.6 grams of diglyme by mechanical stirring. To this mixture, BTDA (32.2 grams) was added and stirring was continued for at least four hours to insure the complete reaction of monomers. If the polyamic acid tended to precipitate after an appreciable viscosity had been reached, small amounts of ethanol were added to redissolve the polymer.

The resin thus prepared was used directly for coating adhesive substrates, as a composite matrix resin in the prepregging of graphite fibers, and for spinning fibers. The resin could be cast as a thin film onto glass and heated to 300°C for one hour to prepare a flexible, self-supporting high-temperature film. The LARC-TPI resin was also precipitated as a solid by pouring into rapidly

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stirring water or methanol; a molding powder was made by drying this solid and heating to complete imidization.

Characterization

The inherent viscosity of the polyamic acid solution was obtained at a concentration of 0.5% in dimethylacetamide at 35°C. Thermomechanical properties of LARC-TPI were obtained by torsional braid analysis (TBA). Glass braids were coated with a 7% polymer solution and preheated to 220°C in air before obtaining TBA spectra. Glass transition temperatures (T_g s) of various films, composites, and moldings were measured by thermomechanical analysis (TMA) on a DuPont 943 Analyzer in static air at a heating rate of 5°C/min. Thermograms of films were obtained by thermogravimetric analysis (TGA) by heating at a rate of 2.5°C/min. in static air. Melt-flow properties of LARC-TPI molding powder were observed by use of a parallel plate rheometer accessory with the DuPont 943 Thermomechanical Analyzer in static air at 5°C/min.

Adhesive Bonding

A LARC-TPI adhesive scrim cloth was prepared for bonding 2.54 cm wide 6Al-4V titanium adherends cleaned by the Pasa Jell 107 acid surface treatment. The resin was coated onto a 112 E-glass (A-1100 finish) cloth and heated for one hour at 100°C and 1/2 hr. at 200°C after each coat. Enough coats were applied to produce a final cloth thickness of 0.25-0.31 mm. Single lap-shear specimens were prepared by sandwiching the cloth between primed adherends using a 1.27 cm overlap. The specimens were bonded as follows:

- (1) RT to 325°C at 7°C/min., apply 1.38 MPa (200 psi) at 280°C,
- (2) Hold 5 min. at 325°C, and
- (3) Cool under pressure.

Lap shear tests were performed on a TT-6 Instron Testing Machine according to ASTM D-1002.

Film Laminating Process

LARC-TPI was used as an adhesive to bond thin polyimide Kapton[®]H-film measuring 0.025-0.076 mm in thickness. The Kapton film was cleaned with ethanol, primed with a thin coat of LARC-TPI adhesive resin, and gradually heated in air to 220°C for one hour to remove excess solvent and complete imidization. The primed film was then thermoplastically bonded to another primed or unprimed sheet of Kapton film as follows:

- (1) Place sandwich between platens preheated to 232°C,
- (2) Apply 0.35-2.07 MPa (50-300 psi),
- (3) Heat to 343°C and hold 5 min., and
- (4) Cool under pressure.

Thin film laminates were also prepared by placing a self-supported adhesive film of LARC-TPI staged to 220°C between two sheets of Kapton film and proceeding according to the above process. Metal-containing laminates were made by placing conductive metal sheets or foils between primed sheets of Kapton and bonding as described above.

Preparation of Molded Discs

Molded discs were prepared from LARC-TPI powder which had been imidized at 220°C. Approximately 20 grams of molding powder was placed in a 5.72 cm diameter steel mold and cured by the following cycle:

- (1) Heat to 200°C in an open mold,
- (2) Insert mold top and apply 6.89 MPA (1000 psi),
- (3) Heat to 260°C, and
- (4) Gradually cool under pressure.

Fracture toughness testing of the 0.16 cm thick discs was performed at the Naval Research Laboratory, Washington, D.C. Round compact tension test specimens were prepared from the LARC-TPI discs and tested for G_{Ic} (the opening-mode strain

energy release rate) (6). Flexural strength and Izod impact strength were measured according to ASTM D-790 and D-256, respectively.

Fabrication of Composites

Diluted LARC-TPI resin (5% solids) was applied as an initial coat to drum-wound Celion 6000 graphite fibers in an effort to ensure good wetting of the fibers. The regular resin at 15% solids content was applied to the fibers in three subsequent coats. The prepreg was air-dried on the rotary drum, cut into 7.6 cm by 17.8 cm segments and stacked into a 12-ply unidirectional preform. The preformed billet was heated under full vacuum from room temperature to 204°C and held for four hours. Upon cooling to room temperature under vacuum, the consolidated billet was placed in a matched-metal mold and laminated in a hydraulic press according to the following cycle:

- (1) RT to 335°C at 5°C/min.,
- (2) Apply 4.14 MPa (600 psi) at 200°C,
- (3) Hold 1/2 hour at 335°C, and
- (4) Cool under pressure.

Mechanical tests on the composite were performed on an Instron Testing Machine TT-6. Flexural strength and modulus was determined according to ASTM D-790. Short beam shear strength was determined using a span-to-thickness ratio of 4.

Spinning of Fibers

Fibers were spun from a 15% solids LARC-TPI polyamic acid solution into a methanol coagulation bath at room temperature. Approximately 0.69 MPa (100 psi) nitrogen was used to pressurize the solution through a teflon spinneret with an orifice measuring .15 mm in diameter. Yellow fibers were spun onto a 7.62 cm diameter teflon roller rotating at 25 rpm which was located in the coagulation bath. The

fibers were imidized by soaking for 24 hours in a 50/50 pyridine/acetic anhydride solution and were then dried either in air or in a forced air oven at 150°C. Single-fiber tensile properties were measured at room temperature.

RESULTS AND DISCUSSION

Resin Chemistry and Properties

LARC-TPI was synthesized according to the reaction scheme shown in Figure 1. Reaction of the monomers in the ether solvent diglyme yielded a highly viscous polyamic acid which had an inherent viscosity of 0.70 dl/g. The thermal imidization of this polyamic acid resin resulted in a linear high molecular weight polyimide which could be processed as a thermoplastic due to its unique molecular structure.

According to the TBA spectrum in Figure 2, LARC-TPI exhibited a relatively low T_g of 229°C after thermal imidization at 220°C in air. Upon further heating at 3°C/min. to 350°C, the polymer had a T_g of 266°C as demonstrated by the TBA spectrum in Figure 2 on cooling from 350°C. LARC-TPI can be processed after imidizing at a reasonably low temperature and then heated further to increase its final use temperature.

LARC-TPI is a very thermo-oxidatively stable polymer as evidenced by the thermograms shown in Figure 3. The dynamic TGA curve obtained on film heated to 300°C in air showed essentially no weight loss prior to 400°C. Weight loss of the material did not exceed 2-3% after isothermal aging at 300°C for 550 hours.

Because it is processable as a thermoplastic after imidization and thermally stable at high temperatures, LARC-TPI shows potential for a variety of applications (Table 1).

Structural Adhesive Properties

LARC-TPI was recently a candidate for the large-area bonding of an experimental graphite composite wing panel in the NASA-Supersonic Cruise Research (SCR) Program, Figure 4. The materials in this program were screened as structural adhesives on both titanium and polyimide/graphite composite adherends. Preliminary bonding results on LARC-TPI were generated both in-house and in studies at Boeing Aerospace Company (NASA Contract NAS1-15605). LARC-TPI exhibited excellent lap shear strengths on titanium at room temperature; and the elevated temperature strengths were above minimum strengths set by the SCR program. The increase in bond strength after aging at 232°C was indicative of a post-cure effect which is characteristic of LARC-TPI adhesive. Although lap-shear specimens were not post-cured after bonding in the present study, a moderate post-cure would advantageously increase the T_g and would therefore increase the use temperature of the adhesive.

The ability of LARC-TPI as a structural adhesive in bonding polyimide/graphite and polyimide/glass composites has been demonstrated and reported elsewhere (3). Composite and composite-to-titanium lap shear strengths averaged approximately 20.7 MPa (3000 psi), but failures occurred in the composites rather than in the adhesive.

Film Laminating

A need exists in the aerospace industry for reliable flexible electrical circuitry that can withstand extreme temperature variations and retain flexibility. Problems to date have been due partially to the presence of voids in film laminates caused by volatiles generated by the adhesive and/or the inherent rigidity of some adhesives. Because it is both flexible and imidized prior to bonding, LARC-TPI shows much potential as a high-temperature adhesive for laminating large areas of polyimide film.

A film-laminating process has been developed whereby films primed with a thin coat of LARC-TPI adhesive are bonded together using temperature and pressure

(Figure 5). As an alternate process, LARC-TPI polyamic acid adhesive film may be imidized by heating prior to being sandwiched between polyimide film. When using either process to produce flexible circuits, a conductive metal may be interposed between layers of the polyimide film. Metal-containing laminates have been made using aluminum, brass, copper and stainless steel sheets or foils.

Large-area polyimide film laminates were made which varied in size from 77.4 cm² to 645 cm². A laminate was also prepared using 8 plies of polyimide film. Larger areas and thicker laminates can be made using the process in Figure 5 as long as the entire laminating area receives an even distribution of temperature and pressure.

In this study, over forty Kapton film laminates were prepared, all of which were clear yellow, flexible, and 100% void-free. Conventional T-peel (ASTM D-1876) and 180°-peel (ASTM D-903) testing of the film laminates was attempted, but no data was generated on the adhesive. Failures occurred due to tearing of the Kapton film indicating that the strength of the LARC-TPI exceeds that of the Kapton film.

In addition to peel strength, the success of a flexible film laminate for circuit applications is determined by its ability to withstand a dip in hot solder. Most laminates will blister (due to the adhesive) after just a few seconds. LARC-TPI laminates, however, have withstood a full 10 second dip in 288°C solder without blistering.

Moldings

Molded discs were prepared from LARC-TPI molding powder by heating to 260°C under 6.89 MPa (1000 psi) pressure. Properties of the moldings are listed in Table 2. Discs measuring 5.72 cm in diameter and 0.16 cm in thickness had a density of approximately 1.40 g/cm³. Discs were flexible but low in T_g when heated only to 260°C during molding with no post cure. Higher T_gs in the range of 275°C have been achieved for moldings pressed at higher temperatures.

LARC-TPI moldings used for measuring flexural strength were in the form of 12.7 cm x 1.27 cm x 0.64 cm bars, and a span of 10.2 cm was employed. The deflection to break for these specimens was nearly twice that displayed by commercial polyimides. Data on LARC-TPI moldings was supplied by R. T. Traskos, Rogers Corporation, Rogers, Connecticut 06263.

Composites

Once imidized, conventional linear polyimides ordinarily do not have enough flow during molding to be used as composite matrix resins. Because of its low T_g and unique meta-oriented molecular structure, however, LARC-TPI has the necessary flow properties and has demonstrated the ability to form composites with graphite fibers. The cure cycle used to mold B-staged LARC-TPI/Celion 6000 billets into composites is displayed in Figure 6. The 12-ply laminates press-molded by this cycle had a final thickness of 1.73 mm. Cross-sections of the laminates were examined by an optical microscope and found to have no lines of demarcation between the resin and fibers.

Properties of the LARC-TPI/Celion 6000 composites are listed in Table 3. An average of two specimens each was used to obtain flexural strength and modulus with a $\pm 5\%$ variation in the data. The short beam shear strength represents an average strength of six specimens with a $\pm 10\%$ data variation. The low value for flexural modulus may be attributed to the high resin content (40.4% by weight) of the composite.

Films and Fibers

Linear polyimides are a prime choice for films and coatings in aerospace applications due not only to their thermal stability, but because of their toughness and flexibility (10). Tough and flexible films were easily made from LARC-TPI amic acid solution cast onto glass plates. Properties shown in Table 4 are characteristic of films air-cured 1 hour each at 100°, 200°, and 300°C having a

final thickness of approximately 0.025 mm (1 mil). Tensile properties (Table 4) reported elsewhere were reasonable in view of the fact that they were recorded on unoriented films having had no specimen thermal treatment following the cure cited above.

The direct wet-spinning of fibers from a LARC-TPI polyamic acid solution has been demonstrated by the procedure outlined in Figure 7. After soaking in a pyridine/acetic anhydride bath for 24 hours, the fibers were found by infrared spectroscopy to be completely imidized. Cross-sections of the fibers were examined by optical microscopy and found to be rounded in shape and to have solid cores. Although the strengths obtained for these fibers are low compared to commercial high-strength fibers, preliminary results show strengths (tenacity = 1.33-1.46 g/d) to be comparable with those of many common textile fibers.

CONCLUSIONS

LARC-TPI is a linear aromatic polyimide which shows potential as a thermoplastic for a variety of high-temperature applications. It is a high molecular weight polymer which is flexible, tough, and thermooxidatively stable at elevated temperatures (300°C). Because of its unique meta-oriented molecular structure, LARC-TPI may be processed as a thermoplastic after imidization and the removal of solvent. After processing, it can be further heated to increase the final use temperature.

LARC-TPI is being evaluated primarily as an adhesive for bonding metals, composites, and films. As a structural adhesive, this new material has demonstrated adequate adhesive strengths for certain advanced aircraft applications. LARC-TPI is also being evaluated as an adhesive in the large-area bonding of polyimide film to produce flexible, 100% void-free laminates for flexible circuit applications. LARC-TPI film laminates have withstood a dip in 288°C solder and are highly resistant to peel forces.

In addition to being used as an adhesive, this resin can be precipitated to form a molding powder and thermoplastically molded at 260°C. Graphite composites have been prepared using LARC-TPI as a matrix resin with promising results.

LARC-TPI also shows potential as a material for making high-temperature films and for spinning high-temperature fibers.

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TABLE 1. APPLICATIONS FOR LARC-TPI

- (1) STRUCTURAL ADHESIVE FOR METALS & COMPOSITES
 - (2) ADHESIVE FOR LAMINATING POLYIMIDE FILM
 - (3) MOLDING POWDER
 - (4) COMPOSITE MATRIX RESIN
 - (5) HIGH-TEMPERATURE FILM
 - (6) HIGH-TEMPERATURE FIBER
-

TABLE 2. PROPERTIES OF LARC-TPI MOLDINGS

DENSITY	1.40 g/cm ³
COMPACT TENSION	1.37 g/cm ³
FRACTURE ENERGY	662 J/m ²
BEGINNING FLOW TEMPERATURE OF MOLDING POWDER	225°C
MAXIMUM FLOW TEMPERATURE OF MOLDING POWDER	255°C
T _g OF DISCS MOLDED AT 260°C (MOLDED AT >260°C*)	225°C 275°C
FLEXURAL STRENGTH	159 MPa (23 KSI)
DEFLECTION TO BREAK*	1.10 cm (0.435 in)
NOTCHED IZOD IMPACT STRENGTH* (.64 cm x 1.27 cm BAR)	21.4 N-M/M (0.4 FT-LB/IN)

*R. T. TRASKOS, SPECIAL PROJECTS GROUP, LURIE R&D CENTER, ROGERS CORPORATION,
ROGERS, CONNECTICUT

TABLE 3. PROPERTIES OF LARC-TPI/CELION 6000 COMPOSITES

RESIN CONTENT	40.4%
DENSITY	1.55 g/cm ³
T _g (NO POST CURE)	244°C
FLEXURAL STRENGTH	1600 MPa (232 ksi)
FLEXURAL MODULUS AT ROOM TEMPERATURE	100 GPa (14.5 x 10 ³ ksi)
SHORT BEAM SHEAR STRENGTH AT 177°C	43 MPa (6.3 ksi)

TABLE 4. LARC-TPI HIGH-TEMPERATURE FILM PROPERTIES

T _g (FILM CURED AT 300°C)	259°C
SOLUBILITY IN ORGANIC SOLVENTS(7)	INSOLUBLE
TENSILE STRENGTH(8)	136 MPa (19.7 KSI)
YIELD STRENGTH(8)	119 MPa (17.3 KSI)
ELONGATION(8)	4.8%
TENSILE MODULUS	3720 MPa (540 KSI)
POLYMER DECOMPOSITION TEMPERATURE	570°C
VOLUME RESISTIVITY(9)	2.0 x 10 ¹⁶ OHMS-CM

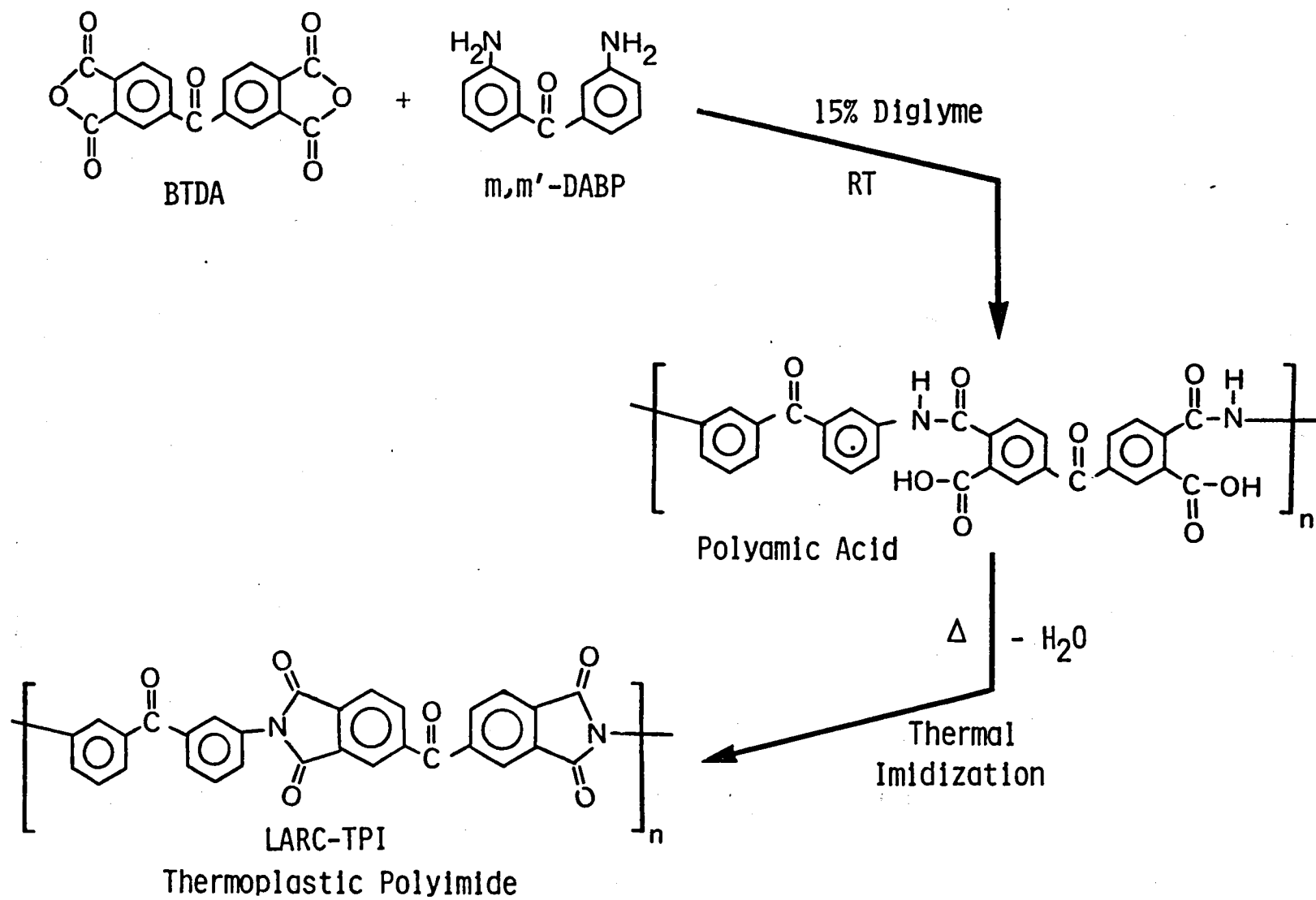


Figure 1. LARC-TPI Chemistry.

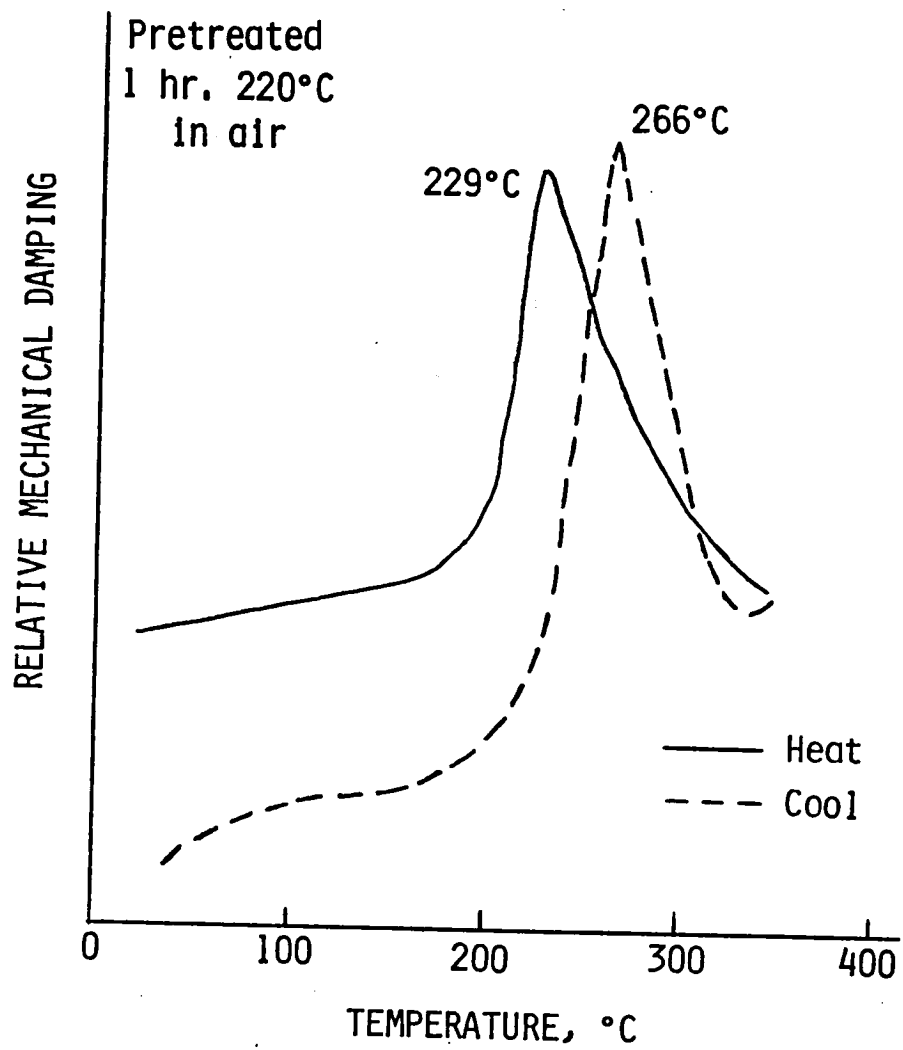


Figure 2. Torsional Braid Analysis of LARC-TPI.

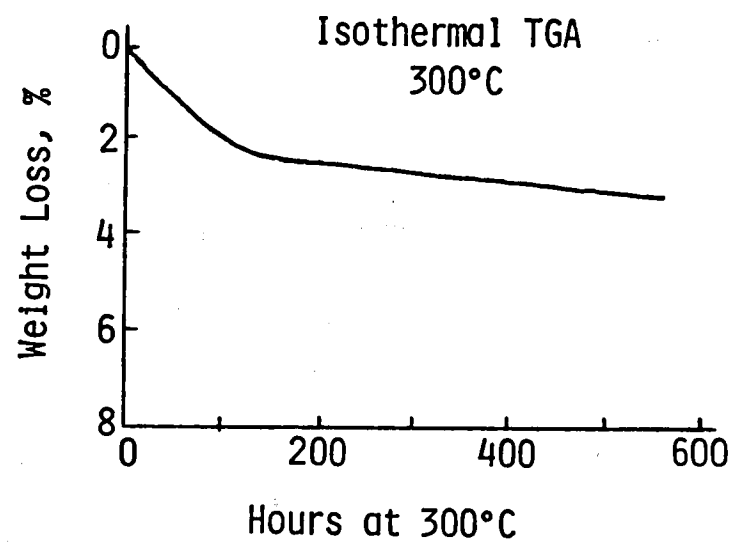
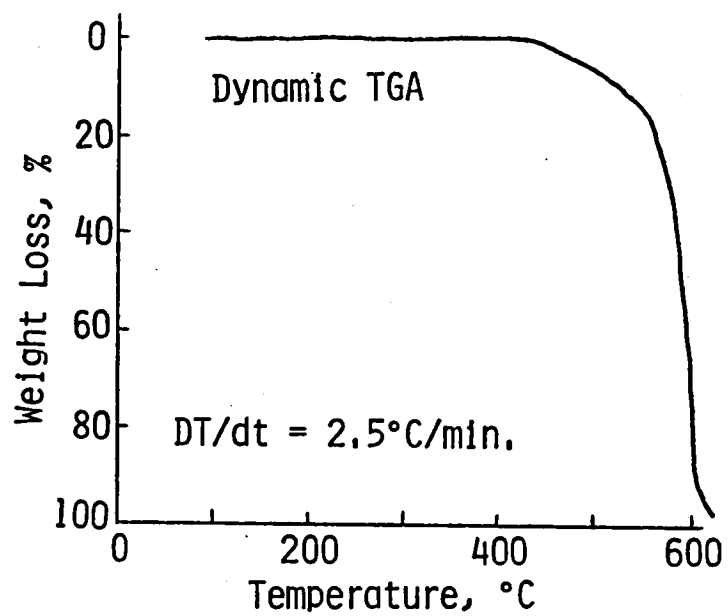


Figure 3. Thermograms of LARC-TPI Film.

DATA FROM	T1/T1 LAP SHEAR STRENGTH, MPa (psi)		T1/T1 LAP SHEAR STRENGTH AFTER 3000 hrs at 232°C MPa (psi)
	RT	232°C	232°C
BOEING AEROSPACE	36.5 (5300)	13.1 (1900)	20.7 (3000)
NASA- LANGLEY	41.4 (6000)	17.9 (2600)	—

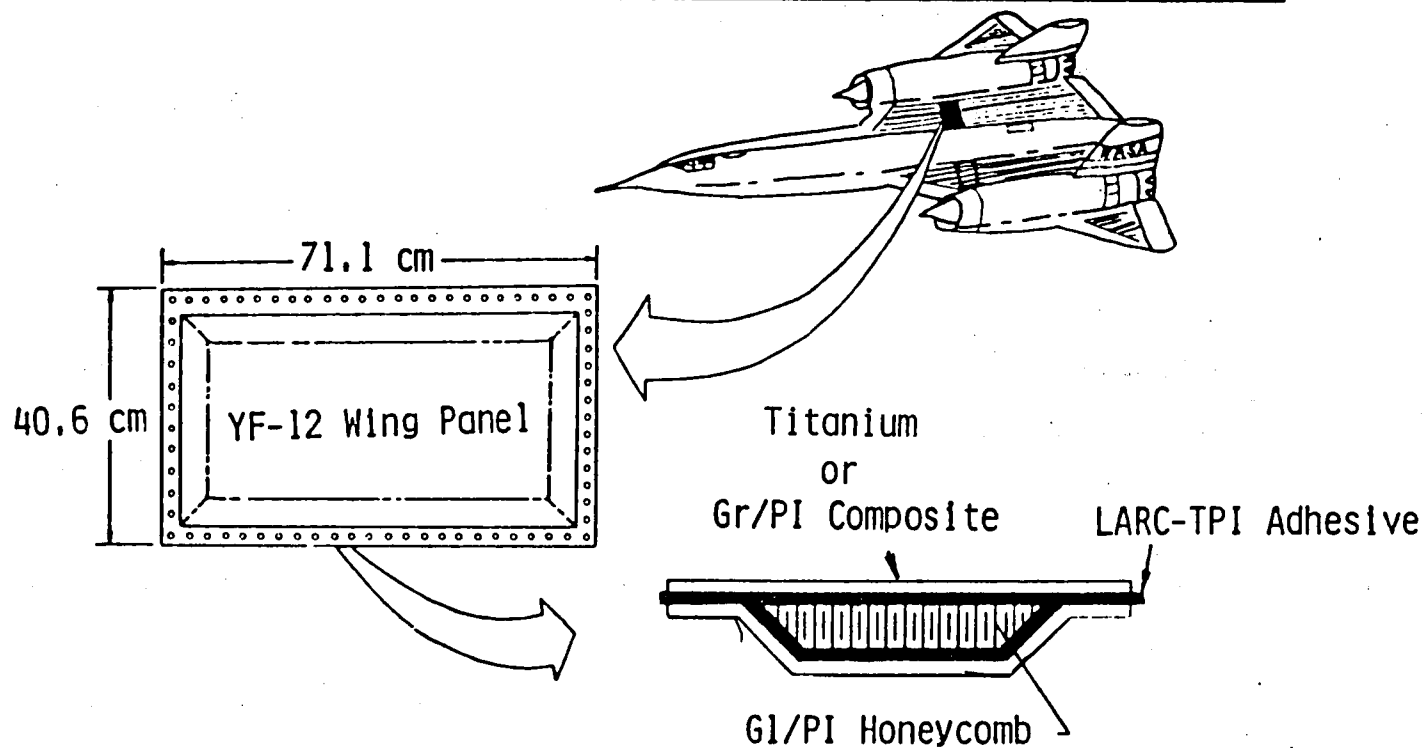


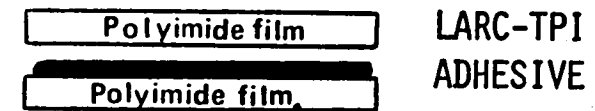
Figure 4. Structural Adhesive Applications of LARC-TPI.

PROCESS:

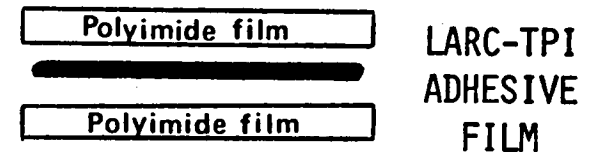
- (1) PLACE SANDWICH BETWEEN
PREHEATED PLATENS (232°C)
- (2) APPLY 0.4-2.1 MPa PRESSURE
- (3) HEAT TO 343°C; HOLD 5 MIN.
- (4) COOL UNDER PRESSURE

RESULTS: CLEAR VOID-FREE LAMINATE

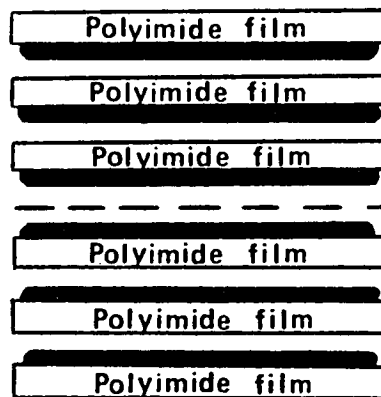
POLYIMIDE FILM LAMINATES



or



MULTI-PLY LAMINATES



METAL-CONTAINING
LAMINATES
(Al, Cu, Steel, Brass)

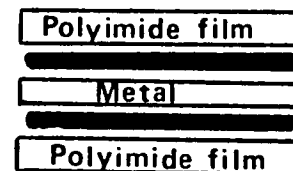


Figure 5. Process for Preparing High-Temperature Film Laminates.

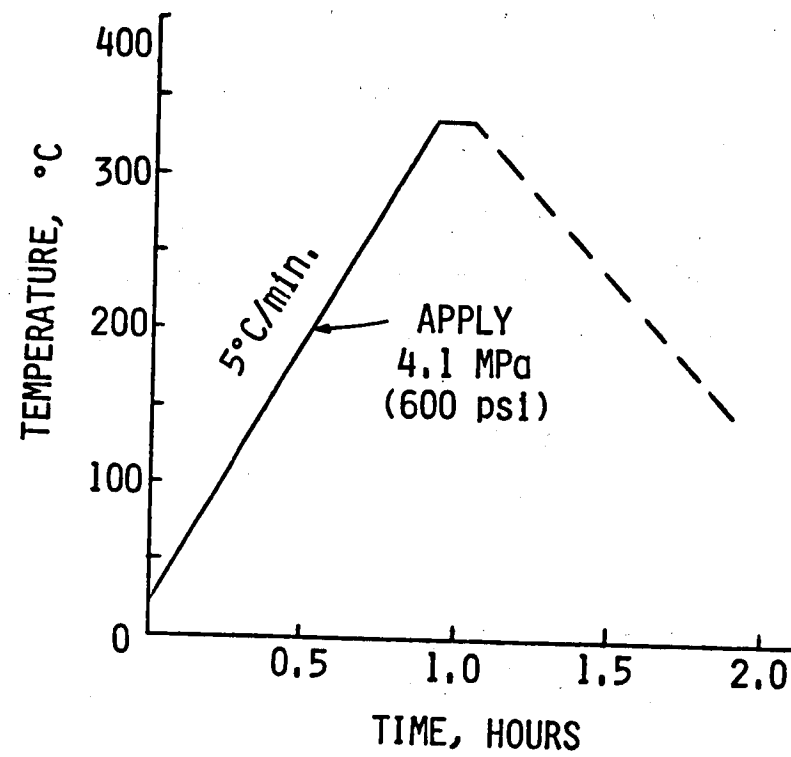
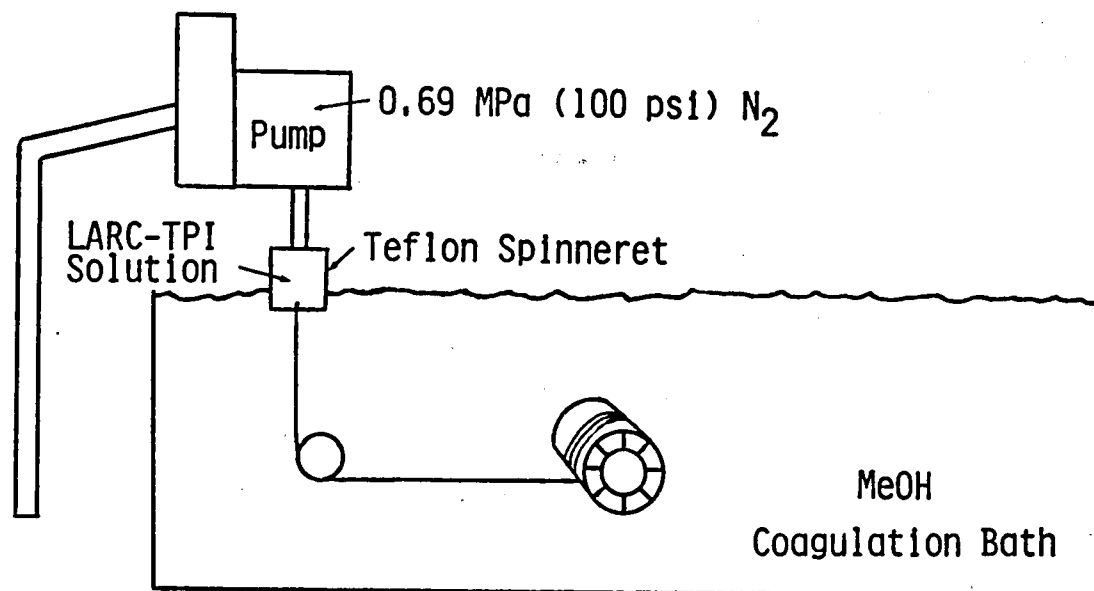


Figure 6. Cure Cycle for LARC-TPI/Celion Composites.



PROCEDURE: (1) Polymer solution pressurized through a 0.15 mm (6 mil) spinneret onto a 7.62 cm roller.

(2) Fibers soaked for 24 hrs in 50/50 pyridine/acetic anhydride.

(3) Fibers dried in air or oven at 150°C.

PRELIMINARY

RESULTS: Fiber Denier 23-38

Fiber Tenacity 1.33-1.46 g/d

Figure 7. Spinning of LARC-TPI Fibers.

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				10. Work Unit No.	
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				13. Type of Report and Period Covered Technical Memorandum	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, DC 20546				14. Sponsoring Agency Code	
15. Supplementary Notes This paper was presented at the 26th National SAMPE Symposium, Los Angeles, CA, April 1981.					
16. Abstract A linear thermoplastic polyimide, LARC-TPI, has been characterized and developed for a variety of high-temperature applications. In its fully imidized form, this new material can be used as an adhesive for bonding metals such as titanium, aluminum, copper, brass, and stainless steel. LARC-TPI is being evaluated as a thermoplastic for bonding large pieces of polyimide film to produce flexible, 100% void-free laminates for flexible circuit applications. The further development of LARC-TPI as a potential molding powder, composite matrix resin, high-temperature film and fiber is also discussed.					
17. Key Words (Suggested by Author(s)) Polyimides Molding Thermoplastic Fiber Adhesive Flexible Circuitry Film Composite				18. Distribution Statement Unclassified - Unlimited Subject Category 27	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 24	22. Price* A02		

